Using pXRF for the Analysis of Ancient Pottery

AN EXPERT WORKSHOP IN BERLIN 2014

Morten Hegewisch
Małgorzata Daszkiewicz
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(eds.)

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THE AIM OF THE SECOND WORKSHOP on the use of portable energy-dispersive X-ray fluorescence (pXRF) organized by the Cluster of Excellence TOPOI was to exchange experiences and discuss the basic requirements for the use of pXRF as a tool for chemical analysis of archaeological ceramics. During two days, 49 participants from eight European countries discussed nineteen lectures, twelve of which are published here as papers presenting research on ceramics and glass of various periods from Bulgaria, Germany, Greece, Rumania, Ukraine, Sudan, Syria and the United Kingdom. The focus was on analysing bulk pottery and on the possibilities of non-destructive determination of chemical composition. The number of chemical elements significant for provenance studies and determinable with sufficient precision and accuracy plays a major role. This was compared with chemical analysis using WDXRF, ICP-MS, NAA. The different examples prove that the chances of positive outcomes depend very much on the individual cases.

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Richard Jones, Louisa Campbell

Testing Composition by pXRF Analysis against Ceramic Shape, Style and Stamp: A Case Study from Samian Found on Hadrian's Wall

Summary

Attributing a source to Roman samian (or Terra Sigillata) pottery has generally been accomplished on the basis of style, decoration, and potters' stamps. However, chemical composition can also play an important role. Our investigation concerns the application of pXRF analysis to situations where sampling for destructive analysis is not possible. This paper reports the results for typologically well-characterized samian, including many stamped sherds, from South Shields fort on Hadrian's Wall. The encouraging results showed that examples of samian ascribed to a particular production center had a uniform, recognizable composition and that comparison with published WD-XRF data gave a provenance assignment that was in agreement with expectations.

Keywords: pXRF; Roman pottery; samian; Gaul; slip

Die Zuordnung römischer Terra Sigillata zu ihren Produktionsorten geschah allgemein auf der Grundlage von Stil, Dekor und Töpferstempeln. Aber auch die chemische Zusammensetzung kann eine wichtige Rolle spielen. Unsere Untersuchung betrifft die Anwendung der pRFA bei Situationen wo eine Probennahme für nicht zerstörungsfreie Analyse unmöglich ist. Dieser Beitrag liefert Ergebnisse für typologisch sicher bestimmte Terra Sigillata, einschließlich vieler gestempelter Scherben, vom South Shields Fort am Hadrian-Wall. Die ermutigenden Ergebnisse zeigten, dass bestimmten Produktionszentren zugeschriebene Beispiele von Terra Sigillata eine einheitliche, erkennbare Zusammensetzung hatten und der Vergleich mit publizierten WD-RFA-Daten eine Herkunftsbestimmung ermöglichte, die in guter Übereinstimmung mit der Erwartung war.

Keywords: pRFA; römische Keramik; Terra Sigillata; Gallien; Glanztonüberzug

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R. J. thanks Ken Grainger and John Hurley at Niton UK for discussions about calibration algorithms, and Gerwulf Schneider and Philippe Sciau for discussions of the results.

1 Introduction

In addition to the contributions to this volume, there have been several reports in the recent archaeological science literature about the application of portable XRF (pXRF) to pottery from archaeological contexts to resolve issues of the pottery's identity, technology and especially origin. For the last of these issues, the reports on prehistoric ceramics – clay tablets found at Hattuša (Boğazköy) and Tell el Amarna,[1] Early-Middle Bronze Age pottery on Cyprus[2] and Chalcolithic pottery in Turkey[3] – have presented encouraging results, while at the same time these studies have identified and characterised some of the limitations that are inherent in the analysis of ceramic surfaces. Statements can indeed be made about origin, generally in the form of associating samples of common composition to a common origin; there can be no claim that the output of pXRF is able to provide more precise and sophisticated information about origin. Rather than replacing the systematic high-quality, multi-element analysis of bulk samples that the destructive techniques, such as WD-XRF, NAA and ICP-ES and ICP-MS, can give, the role of pXRF at present should be seen in providing rapid, and if necessary *in situ*, analysis on a quantitatively larger scale than is usually possible when employing destructive techniques. pXRF thereby presents a broad, objective dimension of information which may set the questions that can be tackled by destructive techniques with access to their associated large databases. Further, the non-invasive and portable character of the technique has great potential for realising the latent research potential of collections under curatorial care.

This paper concerns samian ware (Terra Sigillata) which remains one of the most important and widely studied class of fine ware pottery in the Roman world. As outlined more fully below, there is a wealth of information about its shape and style and much is known about where and how it was made,[4] whilst more research work focusses on the broader social and economic aspects of this ware.[5] The significance of the frequent presence of potters' stamps on samian ware is well explored and understood.[6] Nevertheless, there are instances in which the identity of samian, that is, its assignment to a

1 Goren, Mommsen, and Klinger 2011.
2 Frankel and Webb 2012.
3 Forster et al. 2011.
4 Stanfield and Simpson 1958.
5 Fulford 2013.
6 Hartley and Dickinson 2008–2012.

particular workshop, is uncertain or ambiguous because of the absence of decoration or stamp; it may also be in poor condition. The clear role that chemical analysis can play in this situation led us to investigate samian recovered from various excavations along the Antonine Wall and its vicinity in Scotland.[7] We selected pXRF as the most appropriate analytical technique because all of the assemblages are now acquisitioned in museums that were able only to grant permission for non-destructive work, and, crucially, there were two methodological factors favouring this approach: first the fine textured fabric, and second the characteristic way that samian fractures so often gives a flat, clean break which is suitable for a surface analysis.

In the first phase of our programme of pXRF we analysed samian[8] from four forts along the Antonine Wall – Old Kilpatrick, West Dunbartonshire[9]; Balmuildy, Glasgow[10]; Cadder, East Dunbartonshire; and Bar Hill, East Dunbartonshire[11]. With that data we were able to resolve specific questions regarding the samian from two forts to the south of the Wall of mainly Flavian date[12] namely Castledykes, South Lanarkshire[13] and Loudon Hill, East Ayrshire. At these two forts where much of the samian was undiagnostic and unstamped, two chemical groups were defined: the examples of Flavian date consistently belonged to one group, likely of South Gaulish origin, and a smaller number which joined all the examples from the Antonine Wall whose sources were Lezoux and one other centre of production in Central Gaul.

In this paper we report the results of an internally more controlled exercise, based on well-studied samian from South Shields fort on Hadrian's Wall in northern England.[14] This pottery was selected to include samian that was confidently assigned on the basis of decorative style and in many cases the stamp to different production regions within Gaul. Knowing, as will be explained below, that these regions can be discriminated chemically, our purpose was to establish whether our methodology could yield results that were in accordance with the expectations based on published stylistic/stamp evidence[15] and then as a further check to compare on a qualitative basis our data with that obtained by WD-XRF for the same candidate production centres. A further aim was to add value by analysing the red slip as well as the fabric to determine whether the former's composition was characteristic of the production site in the same way that the body's composition should be. The main effort so far has been in characterising this slip from La Graufesenque and other centres in South Gaul[16] and in particular demon-

7 Jones and Campbell 2016.
8 Now stored in the Hunterian Museum, University of Glasgow.
9 Miller 1928.
10 Miller 1922.
11 Macdonald and Park 1906; Robertson, Scott, and Keppie 1975.
12 1st century AD.
13 Robertson 1964.
14 Dore, Greene, and Johns 1979.
15 Dore, Greene, and Johns 1979; Hartley and Dickinson 1979.
16 Sciau, Languille, et al. 2005; Sciau, Relaix, et al. 2006; Sciau, Sanchez, and Gliozzo 2020.

Fig. 1 Photographs of selected stamps.

strating that the clays of the slip and body were very likely not the same;[17] similar views are emerging about the black gloss on Attic vases.[18]

To put samian ware briefly into context, this class, based on vessel forms and potters' stamps (Fig. 1), plays a fundamental role in dating the Roman presence especially in Rome's frontier regions.[19] But over the last few decades there has been increasing interest in the production aspects of samian. Fülle[20] has explored the internal organisation of the industry at Arezzo, and excavations at numerous production sites,[21] for example at La Graufesenque,[22] have revealed the procedures in making and firing samian; these sites have offered plentiful material for chemical characterisation studies. The standardised technology adopted across Rome's northern provinces in the production of samian – the use of a usually fine-textured, pale coloured, low to medium calcareous clay which was then well fired – provides optimal conditions for such characterisation studies. Because many of the production centres can be reliably differentiated chemically, the role of chemical analysis in samian studies has been important in acting as an objective means of determining origin especially in those cases, which may not be infrequent, where the visual characteristics of the fabrics as set out in *The National Roman Fabric Reference Collection*[23] may be ambiguous or indecisive. Three European laboratories in particular have built up large databases of samian chemical compositions, all using conventional wavelength-dispersive X-ray fluorescence (XRF) spectrometry: Berlin[24], Lyon[25] and Fribourg[26]. One significant application of these databases is Picon's study of samian from

17 Picon 1997.
18 Aloupi-Siotis 2020.
19 E.g. Hartley 1972.
20 Fülle 1997.
21 Tyers 1996.
22 Genin and Vernhet 2002.
23 Tomber and Dore 1998.
24 Schneider 1978.
25 Picon, Vichy, and Meille 1971.
26 Maggetti 1981.

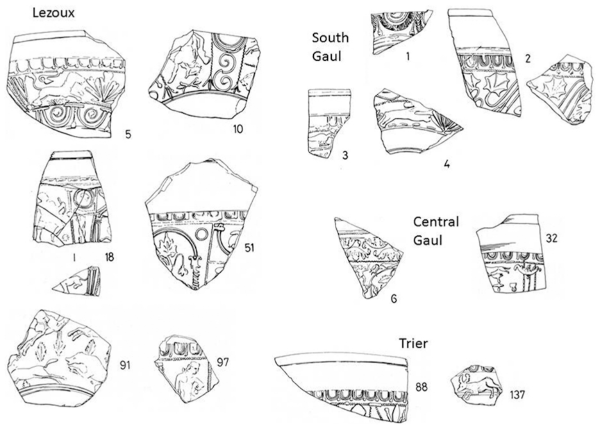

Fig. 2 Illustrations of selected sherds analysed (from Dore, Greene, and Johns 1979, scale 1:2).

the Roman fort at Haltern, showing that Lyon and Pisa, rather than Arezzo were the main suppliers to this fort.[27] Analysis by neutron activation of examples of samian bearing the stamp of Ateius found at Lyons showed decisively that they were products of the Lyon area rather than of this master potter's base at Arezzo;[28] it was inferred that Ateius had established a workshop in this part of Gaul.

2 Material

At the eastern end of Hadrian's Wall lies South Shields fort overlooking the River Tyne. Founded around AD 120, it later became the maritime supply fort for Hadrian's Wall, and was occupied until the Romans left Britain in the 5th century. Of the large assemblage of samian which has been published by Dore et al.[29] and the stamps by Hartley and Dickinson[30], 50 samian sherds[31] were selected for analysis. They are listed in Tab. 1 and some are illustrated in Fig. 2 and Fig. 3.

In order to test the reliability of the pXRF results we deliberately selected samples for analysis which could be relatively confidently ascribed to particular production centres and timeframes through alternative techniques. Rheinzabern and Lezoux feature prominently but other centres are represented as well (Fig. 4). The sherds were in good condition; there was an absence of concretion or surface coating resulting from burial or conservation treatment.

27 Schnurbein, Lasfargues, and Picon 1982; see also Greene 1992, 37.
28 Widemann et al. 1975.
29 Dore, Greene, and Johns 1979.
30 Hartley and Dickinson 1979.
31 Now in the South Shields Museum.

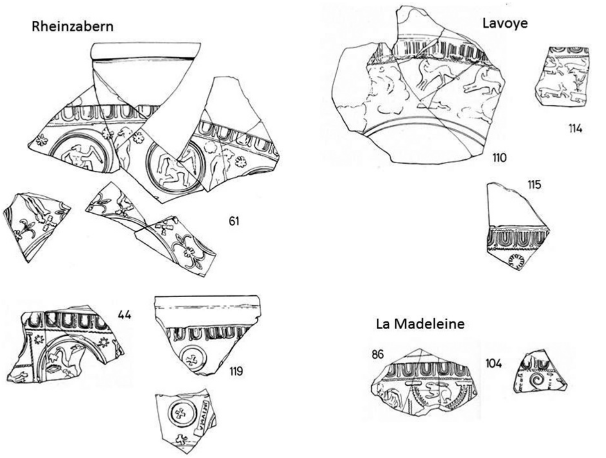

Fig. 3 Illustrations of selected sherds analysed (from Dore, Greene, and Johns 1979, scale 1:2).

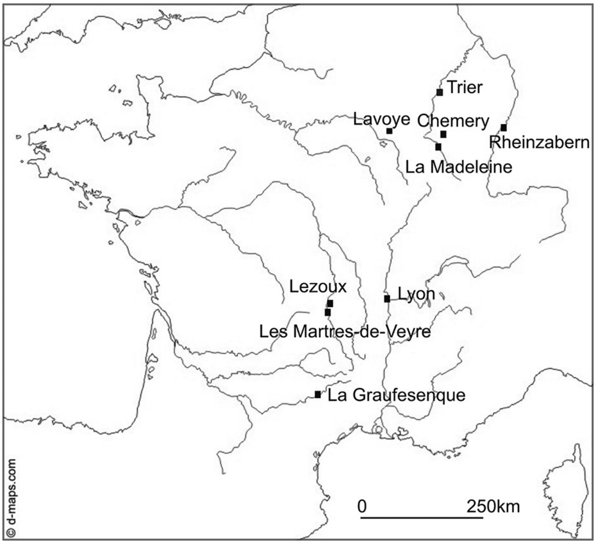

Fig. 4 Map of Gaul showing the samian production centres represented at South Shields Roman fort.

2.1 Method

The analyses were carried out with a portable Thermo Scientific Niton XL3t energy-dispersive XRF instrument with a 50 kV silver X-ray tube and a Geometrically Optimized Large Drift Detector. Each sherd was placed in the lead-lined sample compartment of a Niton-manufactured test stand allowing constant distance and geometry between the X-ray beam and the selected location on the sherd. Three locations were selected, having a fresh break and as flat a surface as possible; the analysis area was estimated at *c.* 10 mm^2. In a few cases the ring base was analysed as well as the fresh break; comparison of the compositions of the ring base in the as-received state and following light sanding of the surface to remove possible surface weathering indicated increases in Fe, Mn and Ca contents in the former of the order of 5%. The count time in each analysis was 60 seconds.[32] Of the instrument's calibration algorithms provided by Niton – Soil, Testall Geo and Mining – Testall Geo gave the preferred results for the range of elements required, but it is nevertheless imperfect when judged against the values of accuracy obtained from analysis of NIST Till 4 and USGS standards (DNC1, AGV2, BCR2 and DTS2).[33] Tab. 2 shows that most elements were underestimated, a major exception being Cr which cannot be reliably determined at concentrations less than 100 ppm; the discrepancy between determined and certified values for this element extends to USGS DNC standard (Cr2 column in Tab. 2). For the four USGS standards simple regression analysis of the certified and determined values gave satisfactory coefficients of determination R^2 values apart from those for V and Cr. Correction factors were determined using the results obtained from the USGS standards whose compositions encompass those of the samian. At least three determinations of each element were examined and found for the most part to lie within 10% of each other, but where one of the determinations deviated by more than 20% it was discounted; analysis of Sr occasionally gave spurious values well in excess of 20%. At least one analysis was made of the slip layer on most sherds.

3 Results

The compositions are given in Tab. 3. Visual examination of the data suggests significant variation in Rb, Zr and Ti contents and this is borne out in the bivariate plots in Fig. 5. 97 with an anomalous high Rb content (244 ppm) is omitted from these plots. When the sample number is replaced by the proposed source based either on vessel form, decoration and where relevant stamp, good correlation is observed (Fig. 6).

- Group 1 encompasses all the examples of samian attributed to Rheinzabern in East Gaul.

- Group 2 contains samian primarily attributed to Lezoux together with one example from Les Martres de Veyre and seven that are loosely defined as Central Gaul.

32 20 seconds each on the main and low energy ranges and light element range; experimentation with longer times gave little improvement in the quality of the data.

33 Wolf and Wilson 2007.

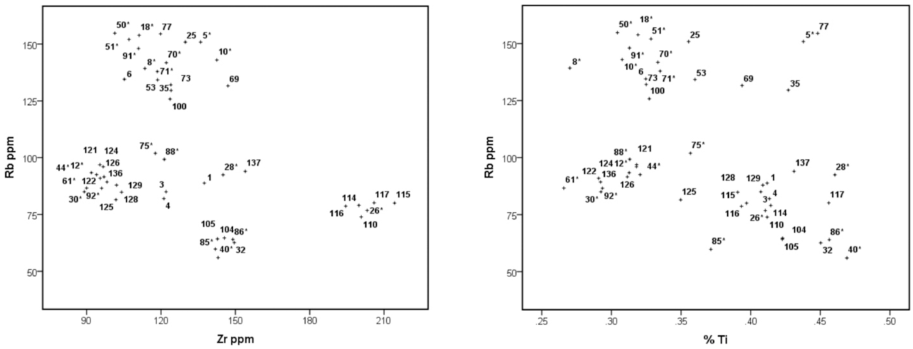

Fig. 5 Plots of Rb-Zr (left) and Rb-Ti (right) contents in samian from South Shields.

– Group 3 is also East Gaul as it comprises four examples from La Madeleine and two assigned stylistically to East Gaul. Dore et al.[34] assign **32** to Central Gaul while noting that its fabric and finish are very similar to that of samian from La Madeleine; its composition however places it firmly in our supposed East Gaul group.

– Group 4 comprises samian from Lavoye and one example, **26**, from Argonne or Trier, again exclusively East Gaul.

Two examples of supposed South Gaul samian, **3** and **4**, lie outside Group 1, as do two examples, **75** and **88**, both stamped that may be from Trier. Two further examples that are assigned to Trier, **28** and **137**, lie well outside Groups 1 and 3.

Multivariate treatment using average link cluster analysis on z score data yields a dendrogram (Fig. 7). There are four significant clusters and several outliers:

– Cluster 1 encompasses members of Groups 1, 3 and 4 and includes Trier **75**.

– Cluster 2 is equivalent to Group 2, i.e. Central Gaul. Anomalous sample **97** belongs weakly to this cluster.

– Cluster 3 consists of Group 1 members – **122**, **124**, 30, **61**, **92**, **136** – and South Gaul **4** and (weakly) Trier **88**. This cluster separates from Cluster 1 owing primarily to higher Ca in the former.

34 Dore, Greene, and Johns 1979.

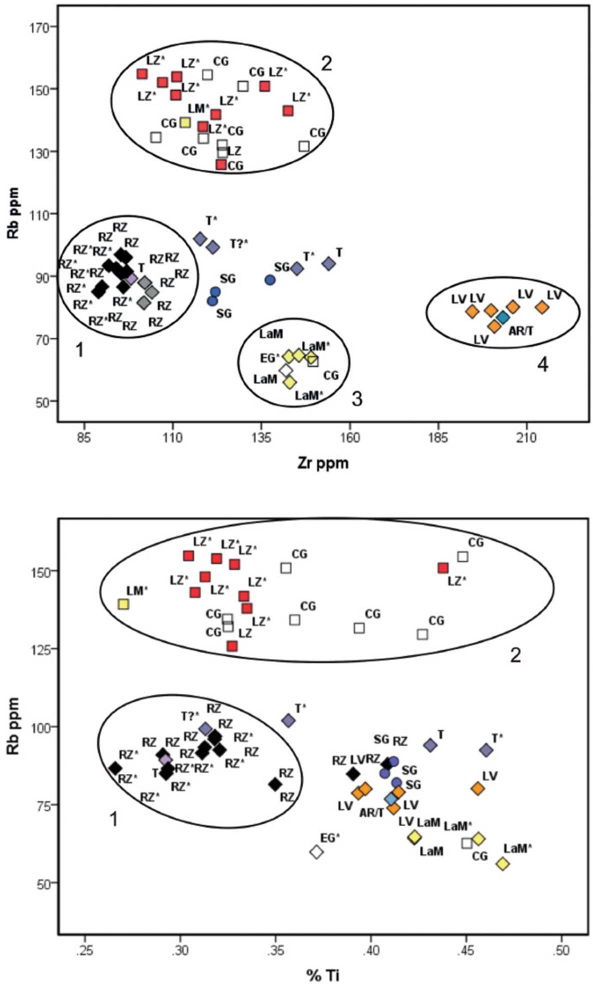

Fig. 6 As for Fig. 5 but the samples are annotated according to projected source based on style and/or stamp. * indicates stamped. *East Gaul diamond*: RZ black, Trier and AR/T blue, Lavoye (LV) orange, La Madeleine (LAM) yellow, E Gaul unclassified no colour. *Central Gaul square*: Lezoux red, Les Martres de Veyre (LM) yellow, C Gaul unclassified no colour. *South Gaul circle*, blue.

The outliers are **28** and **137** (high K), **126** (high Zn), **6** and **10** (high Sr), **3** (high Sr, Ca and Cu) and **110** (low Ca, Mn, Zn and V).

Thus, the cluster analysis has combined most members of the proposed East Gaul Groups 1, 2 and 3 into broad cluster 1 but has separated out a more calcareous East Gaul group. Principal components analysis of the same data set failed to provide a helpful classification since the first two PCs accounted for only 44% of the total variation in composition (PC1 24% dominated by Cr, Ti, –Ca; PC2 20% dominated by Al, Si).

The composition characteristics of the members of Groups 1, 3 and 4 and clusters 1 and 3, all tentatively assigned to East Gaul, are as follows: lower ranges of Ti, Zr, Rb and Sr; wide but on average lower Ca. Trier and La Madeleine have higher Zr and Ti

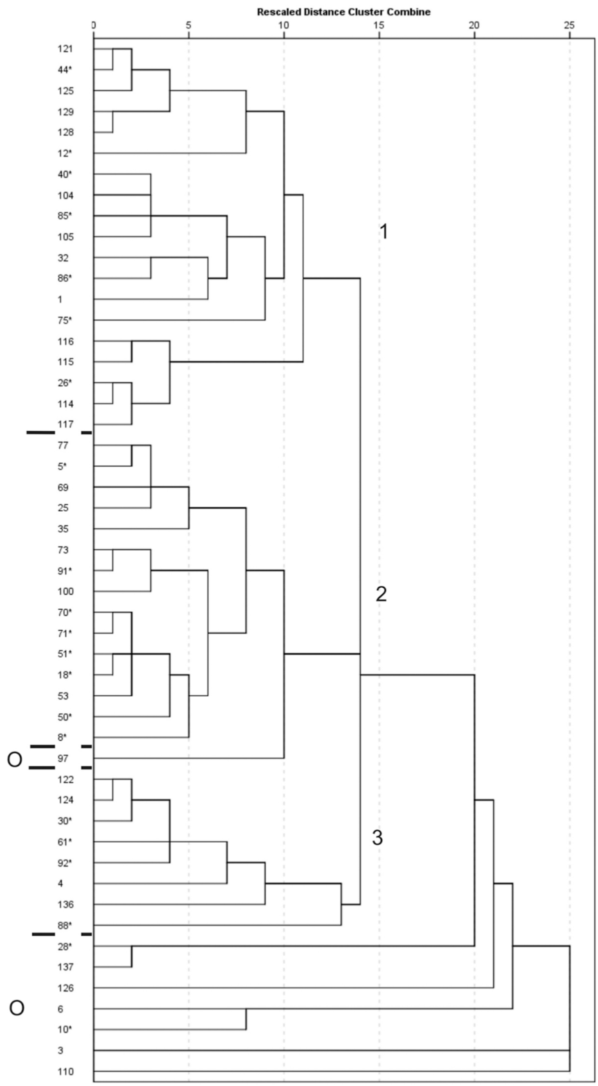

Fig. 7 Average link cluster analysis dendrogram. There are three clusters, 1–3, and Outliers (marked O) 97, 28, 137, 126, 6, 10, 3 and 110.

than Rheinzabern and Trier has notably wide Ca ranges (Fig. 8). Group 2 and Cluster 2, tentatively assigned to Central Gaul, have higher Rb, Sr and Ca contents and wide range of Ti contents.

At this point it can be stated that the samian assigned on the basis of style and/or stamp to particular workshops or regions in Gaul forms coherent chemical groups. There seem to be no discrepancies. With this encouraging picture in mind, the next step is to compare each group with the published data for samian from known work-

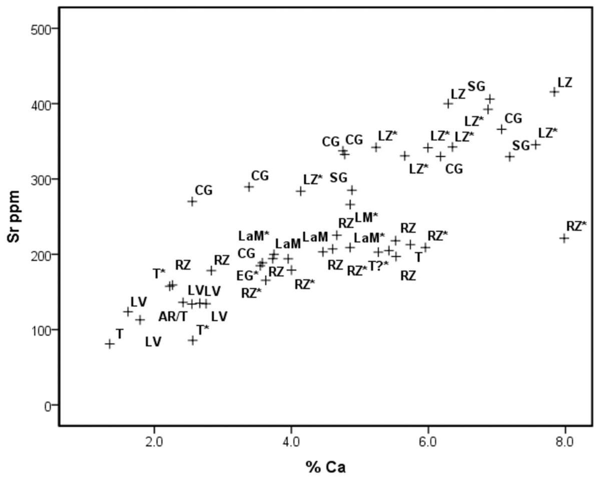

Fig. 8 Plot of Sr-Ca contents.

shops in Gaul, most of it obtained by WD-XRF[35] (Tab. 4). In view of the way the compositions from centres *within* East Gaul – Rheinzabern, Lavoye and La Madeleine – are discernibly different (Figs. 5 and 6), the compositions of several samian reference groups in East Gaul – Chemery, Blickweiler, Trier, Avocourt – have deliberately been included in Tab. 4.

Having corrected the pXRF data according to the figures given in Tab. 2, but treating the Cr comparison as semi-quantitative at best, the first comparison is Group 1 with Rheinzabern. Ca is higher in Group 1 than the Rheinzabern group mean but still within 1 standard deviation; the discrepancy in Mn is slightly larger, but there is excellent agreement in the discriminating elements, Rb, Zr and Ti. Group 2 agrees satisfactorily with the Lezoux group although the former's mean Rb, Sr, Zr and Mn are higher. Despite its very small size, Group 3 agrees adequately with the La Graufesenque group, and this is borne out well in Figs. 9a and 9b although there is total overlap with Lezoux in the Sr-Ca plot (Fig. 9c).

The samples assigned typologically to Trier do not have similar compositions. **75** and **88** share some similarities (e.g. in Rb-Zr) but probably do not have the same source. Neither matches the Trier ICP group (which lacks Rb and Zr determinations) convincingly. **28** and **137** form a pair but in Fig. 8 there is no consistency in the way they associate with a reference group. **136** lies closer to Rheinzabern than anywhere else, and **26** (classed as AR/T) consistently groups with the Lavoye examples in Figs. 5 and 6 and with Chemery and Avocourt in Fig. 9c.

35 Schneider 1978.

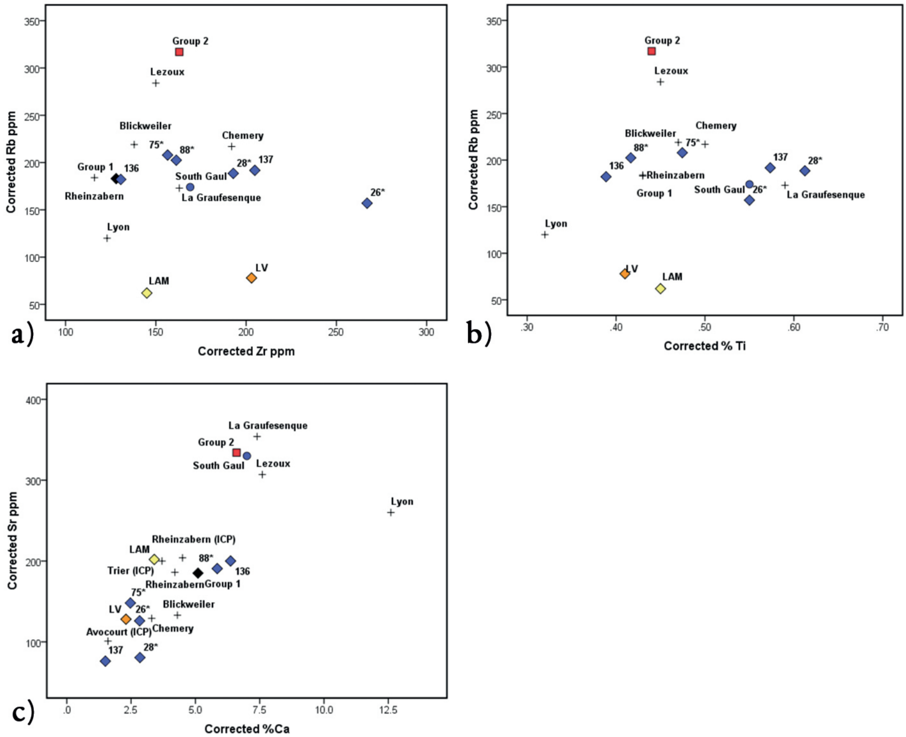

Fig. 9 Plots of *corrected mean* values of (a) Rb-Zr, (b) Rb-Ti and (c) Sr-Ca for Groups 1 and 2, South Gaul group, Lavoye, La Madeleine and individual Trier samples using the coloured symbols as in Fig. 2. The remaining points are the mean values for the Rheinzabern, Lezoux, La Graufesenque, Lyon, Chemery, Blickweiler, Trier and Avocourt reference groups (all indicated with +) as given in Tab. 4. The ranges for each of these groups can be determined from the % coefficient of variation values in Tab. 4.

The Lavoye and La Madeleine samian, which form two separate groups in Fig. 6 left and to a lesser extent in Fig. 6 right, retain their identity in Fig. 9 (which show *mean* values) owing to their lower Rb contents than any of the samian reference groups.

Semi-quantitative analysis of the *slip* layer revealed that it has, as expected, a different composition from that of the body. From the results in Tab. 5 it is apparent that the slip generally has a slightly higher iron content and significantly higher potassium and aluminium but lower calcium contents than the body. On the basis of microprobe analysis of examples of slip from La Graufesenque, Picon[36] used the finding of a K/Al ratio that was double that in the body to propose that the slip was prepared from a less calcareous clay than the body. Working with examples from the same site and from the smaller centre at Montans in the same region, Sciau et al.[37] concurred with this view,

36 Picon 1997, 90.

37 Sciau, Languille, et al. 2005.

demonstrating that the very thin slip,[38] fired at 1020–1080°C, comprised a homogeneous highly sintered layer with uniformly distributed iron and a wide range of quartz crystal sizes.[39]

The K/Al ratio as determined by pXRF is a function not only of the slip and body compositions but also the slip thickness as the X-ray beam penetrates through the slip to a depth of *c.* 100 microns. What pXRF detects therefore is a composite of the major signal arising from the slip coupled with a smaller component from the underlying body. Working on the assumption that the thickness does not vary significantly among the present samples, the results in Tab. 2 and 5 indicate that the three South Gaul specimens, **1**, **3** and **4**, have indeed higher values in the slip than in the body. In the plot of this ratio in the slip and body (Fig. 10) there is one broad cluster with outliers in the form of South Gaul (SG) **3** and Trier **136** and Trier **137**. The South Gaul samples are joined by other samples with ratios greater than 1 but they do not separate from the remaining samples, which themselves form a slight majority, having K/Al ratios less than 1. A potentially important implication is that the latter samples could represent the use of the *same* or similar clay for both slip and body. Turning to another ratio, K/Ca, a high value would point to enriched K in the slip coupled with a low calcareous clay in the slip. The difficulty is that a lower value of this ratio implies a more calcareous clay which could be in either the slip or the body, and to resolve this issue would require further investigation involving analysis by, for example, PIXE. In any case, all the slips, especially the South Gaul examples, have higher K/Ca values than the body except for three Lezoux specimens (**10**, **71** and **100**), one from Rheinzabern (**92**) and three from elsewhere in East Gaul (**26**, **85** and **86**) (Fig. 6).

Examining the distribution of the K/Al and K/Ca ratios among samian from the same production centre or region and bearing in mind the very limited numbers in the Trier and South Gaul groups, the following observations can be made: (1) the relative similarity of slip at Rheinzabern and Lezoux, (2) the relatively higher K/Ca but very uniform K/Al ratios at Trier and (3), as already noted, the higher K/Al values in South Gaul. These observations are compatible with the use of clays selected for the slip that differed from centre to centre, but they may also have a bearing on the level of quality control achieved at each centre.

4 Discussion

This exercise has produced encouraging results. It was designed to test the preliminary findings from our earlier investigation with pXRF analysis of 1st and 2nd C samian re-

38 Estimated at 15 microns thick by Tite, Bimson, and Freestone 1982.

39 Sciau, Relaix, et al. 2006.

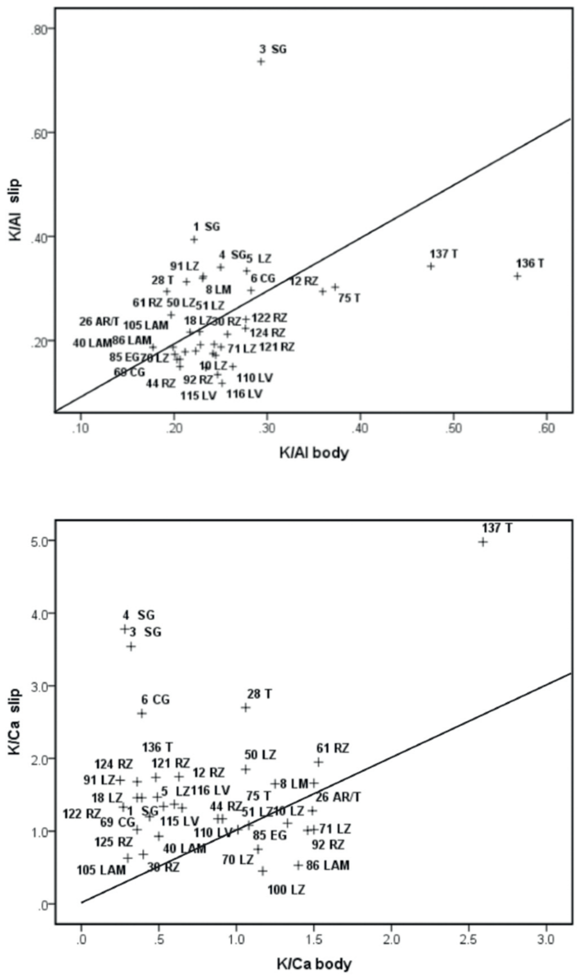

Fig. 10 Plot of (a) K/Al and (b) K/Ca ratios in selected examples of samian slip and body.

covered from forts on and close to the Antonine Wall. The sherds from South Shields on Hadrian's Wall forming the present study were deliberately selected to include examples that were attributable on the basis of decorative style or stamp to a wide range of sources in Gaul. At a first level, the emergent chemical groupings appear to align closely with the sherds' classification according to style and/or stamp, and as such provide a verifiable chemical fingerprint for centres of samian production. At a second level, progress has been made in assigning origin to some of these groupings by relating them to the published chemical (WD-XRF) database of samian production centres. The collective outcome helps validate the use of pXRF as a rapid, non-destructive analytical technique that is capable of interrogating large assemblages of samian sherds. Forthcoming phases of our study are aiming to extend and exploit this capability. Already underway is study of material from excavations of an important Iron Age site at Traprain Law in East Lothian, close to the Roman port at Inveresk which lies to the east of the Antonine Wall. Much of the samian from Traprain Law is either poorly defined typologically or comprises few stamped pieces; in such cases pXRF can expect to take the lead in identifying source regions.

In tandem with the analysis of new material comes the process of addressing a number of methodological issues. One of these is the desirability of introducing a ceramic-specific calibration algorithm to the analysis protocol with Thermo-Niton instruments. Another is to improve the performance characteristics of the kind given in Tab. 2 that should come with the analysis of more ceramic standards; improved performance characteristics will facilitate assessment of the reliability of measurement of important but problematic elements such as Cr. Also assuming increasing importance is the matter of approaching the relationship between the respective compositions determined respectively by WD-XRF and pXRF more directly by analysing by the latter technique either the glass discs prepared for WD-XRF[40] or new samples of samian from the main centres such as Rheinzabern, Lezoux and La Graufesenque.

Finally, this study has contributed to the question of whether the relative technological uniformity of samian production in Gaul extended to the way the slip was prepared. To judge from pXRF analysis, whose limitation that it cannot account for variation in slip thickness has to be acknowledged, the answer would appear to be in the negative. The selection of a less calcareous clay for the slip than the body seems assured at centres in southern Gaul[41], but this was not a uniform procedure since there are examples of samian from elsewhere in Gaul for which the data is more compatible with the use of the same or similar clay for both slip and body. Such a situation is surely consistent with potters adapting their practices to raw materials that were locally available.[42] But more broadly, this line of enquiry raises interesting implications, not necessarily new, regarding quality control[43] and the extent to which, first, the sources of those raw materials changed over time at a given centre and, second, slip preparation was carried out at the individual workshop or centralised level.

40 As was done by Aimers, Farthing, and Shugar 2012 on Mayan ceramics.

41 See relevant results from South Gaul reported by Picon 2002 and Sciau, Vendier, and Dooryhee 2002 and references therein.

42 Despite the significant distance – 12 km – from a source of the red slip used on samian at La Graufesenque, as proposed by Picon 2002, Dannell 2002, 214, makes the important point that the slip may have been refined close to source, thereby reducing considerably its weight and volume during transport to the workshop.

43 See Dannell 2002, fig. 1.

Number in Hartley and Dickinson 1979 (*) or in Dore, Greene, and Johns 1979	Origin (typology/stamp)	Date AD	Stamp (see Hartley and Dickinson 1979)
1	S Gaul	By 90	
3	S Gaul	90–110	
4	S Gaul	90–100	
5*	Lezoux	120–145	Attius
6*	C Gaul	100–120	Austrus
8*	Les Martres de Veyre	120–150	Beliniccus
10*	Lezoux	155–185	Cambus
12*	Rheinzabern	Late 2nd early 3rd	Capitolinus
18*	Lezoux	170-200	Celsianius
25	C Gaul	?	
26*	Argonne/Trier	Mid 2nd	Comus
28*	Trier	Probably Antonine	Craca
30*	Rheinzabern	Late 2nd-early 3rd	Cunissa
32	C Gaul	125–150	
35	C Gaul	?	
40*	La Madeleine	130–160	Genitor
44*	Rheinzabern	180–220	Iulianus
50*	Lezoux	140–160?	Macrianus
51*	Lezoux	150–180	Macrinus
53	C Gaul	?	
61*	Rheinzabern	180–200	Martinus
69*	C Gaul	Antonine	Mercator
70*	Lezoux	150–180	Mossius
71*	Lezoux	160–190	Mox(s)ius
73	C Gaul	?	Mox(s)ius
75*	Trier	180–220	Parentinus

Tab. 1 Samian from South Shields fort analysed by pXRF, arranged according to the number given in Dore, Greene, and Johns 1979 and where stamped (*) by Hartley and Dickinson 1979.

Number in Hartley and Dickinson 1979 (*) or in Dore, Greene, and Johns 1979	Origin (typology/stamp)	Date AD	Stamp (see Hartley and Dickinson 1979)
77	C Gaul	After 150	
85*	E Gaul	c 130–160	Remicus
86*	La Madeleine	130–160	Sabellus
88*	Trier?	Probably late Antonine	Sadiodus
91*	Lezoux	155–190	Secundinus
92*	Rheinzabern	Late 2nd-early 3rd	Severianus
97	Lezoux	Probably early Antonine	Cinnamus?
100*	Lezoux	Mid-late Antonine	Unicus
104	La Madeleine	?	
105	La Madeleine	?	
110	Lavoye	?	
114	Lavoye	?	
115	Lavoye	?	
116	Lavoye	?	
117	Lavoye	?	
119	Rheinzabern	Late 2nd early 3rd	
121	Rheinzabern	Antonine	
122	Rheinzabern	?	
124	Rheinzabern	3rd	
125	Rheinzabern	3rd	
126	Rheinzabern	?	
128	Rheinzabern	3rd	
129	Rheinzabern	?	
136	Trier	Late 2nd early 3rd	
137	Trier	?	

Tab. 1 (Continued) Samian from South Shields fort analysed by pXRF, arranged according to the number given in Dore, Greene, and Johns 1979 and where stamped (*) by Hartley and Dickinson 1979.

	Si	Ti	Al	Fe	Mn	Ca	K	V	Cr	Cr2	Cu	Zn	Rb	Sr	Zr
Till 4 Mean (7)	26.8	3371	6.2	31692	562	6923	9853	231	170	99	219	77	85	120	288
SD	0.14	57	0.1	118	21	178	350	23	17		5.8	3.1	1.3	1.7	2.8
%CV	0.4	1.7	1.7	0.4	3.7	2.6	1.8	9.8	10		2.6	4.1	1.6	1.5	1
Certified		4840		39700	490	8900	27000	53	18	270	237	70	161	109	385
% Accuracy		70		80	87	78	74	23	11	36	92	91	53	91	75
% Accuracy for USGS stds (AGV2, BCR2, GSP2 and DNC1)	93, 83, 94, 77	84, 73, 88, 54	70, 80, 76, 74	97, 100, 86, 88	91, 95, 89, 98	89, 83, 89, 97	90, 79, 90, 74	24, 61, 10, 62	13, 12, 44, 37	36	nd	95, 82, 88, 83	54, 50, 53	88, 98, 91, 99	75, 73, 84, 66
R^2	0.98	0.968	0.986	0.975	0.998	0.988	0.998	0.268	–		nd	0.938	1	0.996	0.998
Correction factor	1.15	1.33	1.33	1.07	0.93	1.11	1.2	0.39		2.78	1.09	1.15	2.04	0.94	1.33

Tab. 2 Performance characteristics of the pXRF analysis: mean, standard deviation, %coefficient of variation and certified values for Till 4 standard. % Accuracy determinations based on data for Till 4 (all elements apart from Si and Al) and USGS standards (all elements except Cu). R^2 values obtained from USGS standards and correction factors determined from USGS accuracy values. All elements expressed as ppm except Si and Al (%). See text for Cr2.

Sherd number in Dore and Gillam 1979	Si	Ti	Al	Fe	Mn	Ca	K	V	Cr	Cu	Zn	Rb	Sr	Zr
1	20.0	0.41	8.6	4.4	730	4.9	1.9	340	42	31	120	89	285	137
3	18.1	0.41	7.5	3.57	1050	6.9	2.2	325	34	134	170	85	406	122
4	18.1	0.41	8.0	2.97	570	7.2	2.0	318	60	40	102	82	330	121
5*	22.7	0.44	7.9	3.68	662	4.1	2.2	316	36	31	123	151	284	136
6	19.4	0.32	6.7	4.02	428	4.9	1.9	276	21	62	108	134	797	105
8*	21.0	0.27	7.9	3.81	791	4.9	2.0	328	39	42	157	139	266	113
10*	22.3	0.31	8.9	4.29	609	3.7	1.6	273	25	53	155	143	1331	143
12*	21.7	0.31	6.4	2.71	802	3.6	2.3	342	74	84	172	93	165	92
18*	19.8	0.32	8.3	3.29	684	6.4	1.9	239	30	48	108	154	342	111
25	24.7	0.36	9.8	3.18	872	3.4	2.3	278	31	50	126	151	289	130
26*	23.4	0.41	8.1	3.12	474	2.6	1.8	343	62	50	96	77	134	203
28*	22.2	0.46	6.5	2.69	665	2.6	4.2	291	72	62	106	92	86	145
30*	16.9	0.29	4.5	2.75	630	4.9	1.8	268	52	48	113	85	209	89
32	23.8	0.45	9.8	3.77	745	3.6	1.7	326	57	37	139	63	185	150
35	23.3	0.43	9.2	4.39	502	2.6	1.8	256	29	35	158	130	270	124
40*	18.7	0.47	7.7	4.22	894	3.8	1.4	344	57	49	168	56	200	143
44*	23.8	0.32	7.8	2.81	581	4.0	1.8	294	64	50	120	93	179	94
50*	17.8	0.3	7.0	2.92	676	5.2	1.9	245	26	30	109	155	342	101
51*	21.4	0.33	10.1	3.28	704	5.7	2.1	267	36	53	117	152	331	107
53	22.6	0.36	10.4	3.49	928	6.2	1.9	257	32	37	133	134	330	119
61*	15.6	0.27	4.7	2.67	793	8.0	1.3	286	47	47	106	87	221	90
69	23.8	0.39	10.3	3.57	602	4.8	2.1	276	36	34	163	132	337	147
70*	21.5	0.33	8.3	3.18	792	6.0	1.9	273	28	38	144	142	341	122
71*	21.3	0.33	8.8	2.86	619	6.9	1.9	271	28	32	141	138	392	119
73	20.2	0.33	7.9	2.79	1103	7.1	1.8	261	31	48	166	132	366	124
75*	20.8	0.36	7.5	3.92	1107	2.2	2.8	322	28	58	172	102	158	118

Tab. 3 The chemical compositions of the samian from South Shields. Si, Ti, Al, Fe, Ca and K expressed as % element, the remainder as ppm element.

Sherd number in Dore and Gillam 1979	Si	Ti	Al	Fe	Mn	Ca	K	V	Cr	Cu	Zn	Rb	Sr	Zr
77	24.0	0.45	8.8	3.76	869	4.8	2.4	286	40	42	143	154	333	120
85*	17.0	0.37	7.6	3.91	1032	3.6	1.6	310	47	39	138	60	189	142
86*	23.0	0.46	9.4	3.8	703	5.4	1.6	283	65	65	152	64	205	149
88*	19.2	0.31	6.3	3.54	1573	5.3	2.8	253	32	49	80	99	203	121
91*	20.5	0.31	8.2	2.83	1123	7.6	1.9	260	29	34	130	148	346	111
92*	19.5	0.29	6.1	3	1200	6.0	1.5	326	49	49	114	87	209	96
97	24.6	0.34	10.3	3.93	612	6.3	2.2	263	27	34	123	244	400	149
100	20.9	0.33	8.5	2.76	1530	7.8	1.8	300	26	53	174	126	416	124
104	20.3	0.42	7.5	3.77	1099	3.7	1.7	289	47	60	169	65	194	146
105	17.9	0.42	6.6	3.9	1393	4.6	1.4	340	52	47	195	64	207	143
110	20.3	0.41	6.2	3.12	553	1.8	1.6	109	61	37	91	74	113	201
114	24.0	0.41	8.0	3.1	589	2.4	2.0	317	65	37	94	79	136	200
115	21.8	0.4	7.0	3.47	506	2.7	1.7	334	52	31	122	80	135	214
116	19.0	0.39	6.2	3.17	509	2.8	1.7	378	50	35	101	79	134	195
117	26.0	0.46	9.8	3.42	522	1.6	2.0	333	67	31	102	80	124	206
121	24.0	0.32	7.6	3.12	658	4.0	1.9	312	57	52	132	97	194	95
122	18.7	0.29	5.5	3.28	519	5.5	1.5	296	52	48	134	91	218	95
124	18.6	0.32	5.8	3.11	559	4.5	1.6	315	59	44	137	96	203	97
125	23.1	0.35	8.2	3.58	675	4.7	1.7	305	55	45	127	81	225	102
126	18.2	0.31	5.2	2.93	589	5.5	1.5	346	59	56	271	92	197	97
128	24.3	0.39	7.3	3.74	609	2.8	1.6	281	75	66	153	85	178	104
129	24.7	0.41	8.3	3.75	515	2.3	1.8	283	84	49	134	88	159	102
136	15.8	0.29	4.8	3.8	1081	5.7	2.7	347	72	47	128	89	213	98
137	23.3	0.43	7.4	2.8	668	1.4	3.5	288	55	69	124	94	81	154

Tab. 3 (Continued) The chemical compositions of the samian from South Shields.

Site	No. of sam-ples	Al	Ti	Fe	Mn	Ca	K	Cr	Rb	Sr	Zn	Zr
Rheinzabern mean	51	9.9	0.43	4.0	0.043	4.2	2.71	125	184	186	123	116
Coefficient of variation (CV%)		1.8	0.17	2.4	13.2	1.8	3.58	4.7	5.5	15	11	9.1
Rheinzabern (ICP) mean	18	10	0.49	3.9	0.047	4.5	2.30	94		204	121	
Standard devia-tion (sd)		0.61	0.07	0.3	0.016	1.3	0.18	15		31	10	
Group 1 mean	12	8.6	0.43	3.3	0.063	5.1	2.04	168	183	185	164	128
sd		1.8	0.06	0.4	0.018	1.7	0.3	32	10	21	51	6
Trier (ICP) mean	4	8.9	0.485	4.6	0.085	3.7	3.85	107		200?	150	
Sd		0.13	0.02	0.6	0.016	0.7	0.32	21		20	7	
Avocourt (ICP) mean	15	9.6	0.64	4.3	0.031	1.6	2.50	87		101	108	
Sd		0.4	0.024	0.2	0.008	0.5	0.12	10		12	11	
Blickweiler mean	12	10.6	0.47	4.8	0.064	4.3	4.77	98	219	133	104	138
CV		1.6	3.17	3.9	3.26	17.1	3.17	6.1	4	9.6	4.1	4.4
Chemery mean	9	10.1	0.5	4.4	0.086	3.3	4.47	95	217	129	92	192
CV		0.8	3.25	1.7	5.9	1.6	1.42	2.5	1.9	3.2	8	5.6
Lezoux mean	15	11.3	0.45	3.7	0.057	7.6	2.83	82	284	307	144	150
CV		1.7	3.3	4.3	16.3	12.1	4.67	5.4	6	13	17	13
Group 2 mean	9	11.5	0.44	3.5	0.075	6.6	2.31	81	317	334	152	163
Sd		1.3	0.05	0.6	0.028	1.5	0.23	11	66	38	24	21
La Graufesenque mean	13	11.9	0.59	4.2	0.056	7.4	3.13	134	173	354	119	163
CV		0.6	1.4	0.7	14	6.1	3.33	2.8	6.2	21	8.9	7.6
Lyon mean	5	7.7	0.32	3.8	0.101	12.6	1.8	76	120	260	81	123
South Gaul this study (mean and range)	3	10.7 (10.0-11.5)	0.55 (0.54-0.55)	3.9 (3.2-4.7)	0.073 (0.053-0.098)	7.0 (5.4-8.0)	2.78 (2.6-3.0)	126 (95-168)	174 (167-181)	330 (268-382)	151 (117-196)	169 (161-135)

Tab. 4 Reference data from Schneider 1978 (WD-XRF) and Hart et al. 1987 (marked ICP); corrected pXRF data for Rheinzabern, Lezoux and South Gaul groups appear in grey highlight. Al to K are % element, the remainder are ppm element; sd standard deviation.

Number and source	Fe %	Ca %	K %	Al%	K/Ca slip	K/Ca body	K/Al slip	K/Al body
1 SG	4.3	2.4	3.48	8.8	1.46	0.39	0.39	0.22
3 SG	4.1	1.9	6.69	9.1	3.54	0.32	0.74	0.29
4 SG	4.1	1.2	4.53	13.3	3.78	0.28	0.34	0.25
5 LZ	4.0	2.4	3.22	9.6	1.34	0.53	0.33	0.28
6 CG	4.7	1.4	3.67	12.4	2.62	0.39	0.30	0.28
8 LM	4.4	1.9	3.12	9.8	1.66	1.50	0.32	0.25
10 LZ	4.5	2.3	2.53	14.5	1.11	1.33	0.17	0.25
12 RZ	3.0	1.9	3.36	11.4	1.75	0.63	0.29	0.36
18 LZ	4.9	1.9	2.79	14.6	1.46	0.36	0.19	0.23
26 AR/T	3.1	1.8	2.25	10.4	1.28	1.49	0.22	0.24
28 T	3.1	1.5	4.16	13.3	2.70	1.06	0.31	0.21
30 RZ	3.4	3.5	2.36	11.1	0.68	0.40	0.21	0.26
40 LAM	4.0	2.5	2.33	12.4	0.93	0.50	0.19	0.18
44 RZ	4.8	1.0	1.23	8.2	1.17	0.88	0.15	0.21
50 LZ	4.3	1.8	3.31	13.3	1.85	1.06	0.25	0.20
51 LZ	4.5	2.5	2.69	12.4	1.08	1.08	0.22	0.23
61 RZ	3.8	1.8	3.58	12.2	1.95	1.53	0.29	0.25
69 CG	5.0	2.0	2.36	14.4	1.20	0.44	0.16	0.20
70 LZ	3.0	2.5	2.51	10.0	1.02	1.01	0.19	0.25
71 LZ	4.1	3.0	3.04	15.8	1.02	1.50	0.19	0.26
75 T	4.6	2.2	3.58	11.8	1.65	1.25	0.30	0.37
85 EG	3.9	2.8	2.09	12.1	0.75	1.14	0.17	0.20
86 LAM	3.8	3.9	2.08	11.6	0.53	1.40	0.18	0.20
91 LZ	3.9	2.5	4.32	13.4	1.70	0.25	0.32	0.23
92 RZ	3.5	2.5	2.58	15.0	1.01	1.46	0.17	0.24
100 LZ	4.0	4.3	1.94	13.1	0.45	1.17	0.15	0.23
105 LAM	4.1	3.0	1.89	10.6	0.63	0.30	0.18	0.21

Tab. 5 Fe, Ca, K and Al contents and K/Ca in the red slip and K/Ca in the body. Instances of the body having a higher K/Ca ratio than the slip are grey highlighted.

Number and source	Fe %	Ca %	K %	Al%	K/Ca slip	K/Ca body	K/Al slip	K/Al body
110 LV	4.8	1.0	1.23	8.2	1.17	0.91	0.15	0.26
115 LV	4.1	1.2	1.52	11.3	1.32	0.65	0.13	0.25
116 LV	3.5	1.2	1.70	14.4	1.37	0.60	0.12	0.25
121 RZ	4.0	1.5	2.68	14.3	1.74	0.48	0.19	0.25
122 RZ	3.5	2.0	2.71	12.2	1.33	0.27	0.22	0.28
124 RZ	3.5	1.7	2.79	11.6	1.68	0.36	0.24	0.28
125 RZ	3.7	2.5	2.50	15.3	1.02	0.36	0.16	0.21
136 T	4.4	2.9	4.21	13.0	1.47	0.49	0.32	0.57
137 T	3.0	0.8	4.16	12.1	4.98	2.59	0.34	0.48

Tab. 5 (Continued) Fe, Ca, K and Al contents and K/Ca in the red slip and K/Ca in the body. Instances of the body having a higher K/Ca ratio than the slip are grey highlighted.

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